

GEORGIA INSTITUTE OF TECHNOLOGY

OFFICE OF RESEARCH ADMINISTRATION

RESEARCH PROJECT INITIATION

Date: 16 March 1973

Project Title: "Fourier Transform System for Nuclear Magnetic Resonance"

Project No: G-33-659

Principal Investigator: Dr. W. M. Spicer

Sponsor: National Science Foundation

Agreement Period: From February 26, 1973 Until September 30, 1974

Type Agreement: Grant No. GP-37138

Amount: \$57,600 NSF Funds (G-33-659)  
57,600 GIT Contribution (G-33-346)  
\$115,200 Total

Reports Required: Final Report

Sponsor Contact Person (s):

Administrative

W. W. Bolton  
Grants Officer  
National Science Foundation  
Washington, D. C. 20550

Technical

Dr. Richard S. Nicholson  
Program Director for Chemical  
Instrumentation  
National Science Foundation  
Washington, D. C. 20550

Assigned to: School of Chemistry

COPIES TO:

Principal Investigator

School Director

Dean of the College

Director, Research Administration

Director, Financial Affairs (2)

Security-Reports-Property Office

Patent Coordinator

Library

Rich Electronic Computer Center

Photographic Laboratory

Project File

Other \_\_\_\_\_

GEORGIA INSTITUTE OF TECHNOLOGY  
OFFICE OF CONTRACT ADMINISTRATION  
SPONSORED PROJECT TERMINATION

Postcard  
N-F  
all

Date: December 3, 1976

Project Title: Fourier Transform System for Nuclear Magnetic Resonance

Grant GP-37138

Project No: G-33-659

No prop. Actions -

Project Director: Dr. W. M. Spicer

OK

Sponsor: National Science Foundation

Effective Termination Date: 3/31/75

Clearance of Accounting Charges: 3/31/75

Grant/Contract Closeout Actions Remaining: none

- ☐ Final Invoice and Closing Documents
- ☐ Final Fiscal Report
- ☐ Final Report of Inventions
- ☐ Govt. Property Inventory & Related Certificate
- ☐ Classified Material Certificate
- ☐ Other \_\_\_\_\_

Assigned to: Chemistry (School/Laboratory)

COPIES TO:

Project Director  
Division Chief (EES)  
School/Laboratory Director  
Dean/Director—EES  
Accounting Office  
Procurement Office  
Security Coordinator (OCA) ✓  
Reports Coordinator (OCA)

Library, Technical Reports Section  
Office of Computing Services  
Director, Physical Plant  
EES Information Office  
Project File (OCA)  
Project Code (GTRI)  
Other \_\_\_\_\_

6-33-659

GEORGIA INSTITUTE OF TECHNOLOGY  
ATLANTA, GEORGIA 30332

CHEMISTRY

November 23, 1976

Dr. Thomas C. Farrar, Director  
Chemical Instrumentation Program  
Chemistry Division  
National Science Foundation  
Washington, D. C. 20550

Dear Dr. Farrar:

A final report on Chemical Instrumentation Grant GP-37138 is enclosed. A JEOL PFT-100 Spectrometer was purchased and has been operational since the fall of 1973. In addition to supplying matching funds for the grant, Georgia Institute of Technology has supplied funds for a technician to operate and maintain the instrument. The JEOL FT NMR has been an invaluable asset to the department and has added greatly to our scientific and educational productivity. The attached report outlines the research projects that have been aided by this instrument and includes lists of papers published, manuscripts submitted, theses completed, and research programs in progress at the Postdoctoral, Ph.D., and M.S. levels. Two reprints of each of the published papers are also included.

On behalf of the School of Chemistry, I would like to express appreciation to NSF for the funds provided for this instrument through grant GP-37138.

Sincerely,

J. A. Bertrand, Director  
School of Chemistry

JAB/mkw

Enclosures

RESEARCH PROJECTS IN CHEMISTRY AT THE GEORGIA INSTITUTE OF TECHNOLOGY  
THAT HAVE BEEN AIDED BY A FOURIER TRANSFORM NUCLEAR MAGNETIC RESONANCE  
SPECTROMETER PURCHASED IN PART WITH FUNDS FROM NATIONAL SCIENCE  
FOUNDATION CHEMICAL INSTRUMENTATION GRANT GP-37138, APRIL, 1973

FINAL REPORT

## I. Published Papers

Published papers of research investigations at Georgia Tech that have been aided by the FT NMR purchased with funds from the National Science Foundation.

1. "The Chemistry of Carbanions. XXVI. The Synthesis of Certain Alkenyl  $\alpha,\beta$ -Unsaturated Ketones," Herbert O. House, Wei C. Liang, and Paul D. Weeks, J. Org. Chem., 39, 3102 (1974).
2. "Some Stereochemical Aspects of the Claisen Rearrangement of Allyl Vinyl Ethers," Herbert O. House, Jacek Lubinkowski, and James J. Good, J. Org. Chem., 40, 86 (1975).
3. "Reactions Involving Electron Transfer. VI. A Stereochemical Test for Anion Radical Intermediates in Additions to Carbonyl Compounds," Herbert O. House and Paul D. Weeks, J. Amer. Chem. Soc., 97, 2770 (1975).
4. "Reactions Involving Electron Transfer. VII. Use of Intramolecular Reactions as a Test for Anion Radical Intermediates," Herbert O. House, and Paul D. Weeks, J. Amer. Chem. Soc., 97, 2778 (1975).
5. "Reactions Involving Electron Transfer. VIII. The Reaction of Trityllithium with Enones," Herbert O. House and Paul D. Weeks, J. Amer. Chem. Soc., 97, 2785 (1975).
6. "The Chemistry of Carbanions. XXVII. A Convenient Precursor for the Generation of Lithium Organocuprates," Herbert O. House, Chia-Yeh Chu, Joyce M. Wilkins, and Michael J. Umen, J. Org. Chem., 40, 1460 (1975).
7. "Use of Lithium Organocuprate Additions as Models for an Electron Transfer Process," Herbert O. House, Accts. Chem. Res., 9, 59 (1976).
8. "Cyclization of Unsaturated Hydroxylamine Derivatives," Herbert O. House, David T. Manning, David G. Melillo, Len F. Lee, O. R. Haynes, and Bruce E. Wilkes, J. Org. Chem., 41, 855 (1976).
9. "A New Synthesis of 2-Alkylpyrrolidines and 2-Alkylpiperidines," Herbert O. House and Len F. Lee, J. Org. Chem., 41, 863 (1976).
10. "The Chemistry of Carbanions. XXVIII. The Carbon-13 Nuclear Magnetic Resonance Spectra of Metal Enolates," Herbert O. House, Ananth V. Prabhu, and William V. Phillips, J. Org. Chem., 41, 1209 (1976).
11. "Perhydroindan Derivatives. XVII. Application of the Reduction-Methylation Sequence to 7-Methoxyhexahydrofluorene Derivatives," Herbert O. House, Roger C. Strickland, and Edward J. Zaiko, J. Org. Chem., 41, 2401 (1976).
12. "Reactions Involving Electron Transfer, 9. Reaction of Lithium Dimethylcuprate with Alkyl Aryl Ketones," Herbert O. House, Ananth V. Prabhu, Joyce M. Wilkins, and Len F. Lee, J. Org. Chem., 41, 3067 (1976).

I. Published Papers (Continued)

13. "Reactions Involving Electron Transfer, 10. The Use of  $\beta$ -Cyclopropyl  $\alpha,\beta$ -Unsaturated Ketones to Detect Anion Radical Intermediates," Herbert O. House and Karel A. J. Snoble, J. Org. Chem., 41, 3076 (1976).
14. "Reactions Involving Electron Transfer. 11. Reactions of Lithium Dimethylcuprate with Diaryl Ketones," Herbert O. House and Chia-Yeh Chu, J. Org. Chem., 41, 3083 (1976).
15. "The Structure of a Substituent Stabilized Thione Methylide," A. J. Arduengo and E. M. Burgess, J. Amer. Chem. Soc., 98, 5021 (1976).
16. "Proton Spin Relaxation in the Halomethanes," P. M. Brown, N. R. Krishna, and S. L. Gordon, J. Mag. Resonance, 20, 540 (1975).
17. "Enzymatic Epoxidation of trans,trans-1,8-Dideutero-1,7-octadiene," S. W. May, S. L. Gordon, and M. S. Steltenkamp, Fed. Proc., 35, 1536 (1976).
18. "Formic Acid-Catalyzed Rearrangement of Carotol. X-Ray Crystal Structure of the p-iodobenzoate of One of the Alcohols Formed," L. H. Zalkow, M. G. Glower, Jr., M. Gordon, J. Smith, D. VanDerveer, and J. A. Bertrand, J. Chem. Soc. Chem. Commun., 374 (1976).
19. "Thermodynamics of Acid-Base Equilibria. Ionization of Thiophenol--An Ion Size Effect," C. L. Liotta, E. M. Perdue and H. P. Hopkins, Jr., J. Amer. Chem. Soc., 96, 7981 (1974).
20. "Thermodynamics of Acid-Base Equilibria VI. Ionization of Substituted Pyridinium Ions," C. L. Liotta, E. M. Perdue and H. P. Hopkins, Jr., J. Amer. Chem. Soc., 96, 7308 (1974).
21. "Influence of Solvation Factors on Acidity. Thermodynamics of Hydration of a Series of Meta- and Para-Substituted Phenols," C. L. Liotta, H. P. Hopkins, Jr., and P. T. Kasuida, J. Amer. Chem. Soc., 96, 7153 (1974).
22. "Concerning the Decarbalkoxylations of Geminal Diesters,  $\beta$ -Keto Esters and  $\alpha$ -Cyano Esters Effected by Sodium Chloride in Wet Dimethyl Sulfoxide," C. L. Liotta and F. L. Cook, Tetrahedron Letters, No. 13, 1095 (1974).
23. "Preparation and Purification of 18-Crown-6," C. L. Liotta, H. P. Harris, F. L. Cook, G. W. Gokel, and D. J. Cram, J. Org. Chem., 39, 2445 (1974).
24. "Facile Synthesis of 12-Crown-4 and 15-Crown-5," C. L. Liotta, F. L. Cook, C. W. Bowers, and T. Caruso, Tetrahedron Letters, No. 46, 4020 (1974).
25. "Chemistry of "Naked" Anions I. Reaction of the 18-Crown-6 Complex of Potassium Fluoride with Organic Substrates in Aprotic Organic Solvents," C. L. Liotta and H. P. Harris, J. Amer. Chem. Soc., 96, 2250 (1974).
26. "Chemistry of "Naked" Anions II. Reactions of the 18-Crown-6 Complex of Potassium Acetate with Organic Substrates in Aprotic Organic Solvents," C. L. Liotta, H. P. Harris, M. McDermott, T. Gonzalez and K. Smith, Tetrahedron Letters, No. 28, 2417 (1974).

I. Published Papers (Continued)

27. "Chemistry of "Naked" Anions III. Reactions of the 18-Crown-6 Complex of Potassium Cyanide with Organic Substrates in Aprotic Organic Solvents," C. L. Liotta, F. L. Cook and C. W. Bowers, J. Org. Chem., 39, 3416 (1974).
28. "Stereochemistry of Nucleophilic and Electrophilic Reactions by the Orbital Distortion Technique. Addition Reactions to Enole and Enone Systems," C. L. Liotta, Tetrahedron Letters, 8, 519 (1975); Erratum, No. 21, 1660 (1975).
29. "Stereochemistry of Allylic Nucleophilic Displacement Process by the Orbital Distortion Technique," C. L. Liotta, Tetrahedron Letters, 8, 523 (1975).
30. "Relative Nucleophilicities of "Naked" Anions," C. L. Liotta, E. Grisdale, and H. P. Hopkins, Jr., Tetrahedron Letters, 48, 4205 (1975).
31. "A Quantitative Comparison of Gas and Solution Phase Basicities of Substituted Pyridines," C. L. Liotta, H. P. Hopkins, C. J. Alexander, D. H. Aue, H. M. Webb, and M. T. Bowers, J. Amer. Chem. Soc., 98, 854 (1976).

II. Manuscripts Submitted

Submitted manuscripts of research investigations at Georgia Tech that have been aided by the FT NMR purchased with funds from the National Science Foundation.

1. "The Chemistry of Carbanions, 29. The Nature of the Enolate Formed by Addition of Lithium Dimethylcuprate to Enones," Herbert O. House and Joyce M. Wilkins, J. Org. Chem., 41.
2. "The Chemistry of Carbanions, 30. Stereochemistry of the Metal-Ammonia Reduction of 7-t-Butyl-10-methyl- $\Delta^{1,9}$ -octal-2-one," Herbert O. House and Michael J. Lusch, J. Org. Chem., 41.
3. "The Structure of the Substance C<sub>27</sub>H<sub>38</sub>O Formed by the Base-Catalyzed Self-Condensation of Isophorone," J. Aaron Bertrand, Duncan Cheung, Audrey D. Hammerich, Herbert O. House, Walter T. Reichle, Don Van Derveer, and Edward J. Zaiko, J. Org. Chem.
4. "The Synthesis of  $\omega$ -Bromo Ketones," Herbert O. House, Chia-Yeh Chu, William V. Phillips, Trevor S. B. Sayer, and Cheuk-Chung Yau, J. Org. Chem.
5. "Concerning the Reaction of Organocuprates with 4-tert-Butylcyclohexanone," E. C. Ashby, J. J. Lin, and J. J. Watkins, Tetrahedron Letters.
6. "Concerning the Significance of Product Development Control as an Important Factor in the Reduction and Alkylation of Model Ketone Systems," E. C. Ashby, and S. A. Noding, J. Org. Chem.



## II. Manuscripts Submitted (Continued)

7. "Enzymatic Epoxidation of Trans, trans-1,8-Dideutero-1,7-Octadiene: Analysis Using Partially Relaxed Proton Fourier Transform NMR," S. W. May, S. L. Gordon and M. S. Steltenkamp, J. Amer. Chem. Soc.
8. "Carbanions. XVIII. Spiro Anions from Reactions of 2 and 3-p-Biphenyl-alkyl Chlorides with Cs-K-Na Alloy," J. A. Bertrand, E. Grovenstein, Jr., D. C. Lu, and D. Van Derveer, J. Amer. Chem. Soc.

## III. Theses Completed

Theses completed at Georgia Tech that have been aided by the FT NMR purchased with funds from the National Science Foundation.

### Ph.D. Category

<u>Name</u>	<u>Title of Thesis</u>	<u>Thesis Advisor</u>
A. J. Arduengo	"The Synthesis, Structure and Chemistry of Substituent Perturbed Thione S-Methylides and Dihalothioureas," 1976	Dr. E. M. Burgess

### M. S. Category

R. L. Harris	"Applications of Proton and Carbon-13 Nuclear Magnetic Resonance to the Study of Molecular Translation in Liquid Solution," 1974	Dr. S. L. Gordon
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## IV. Research in Progress

Research in progress at Georgia Tech that is making use of the FT NMR purchased with funds from the National Science Foundation.

### Post-Doctoral Investigations

<u>Name</u>	<u>Title of Research</u>	<u>Advisor</u>
Dr. T. S. B. Sayer	"The Synthesis of $\omega$ -Bromo Ketones"	Dr. H. O. House
Dr. C-Y Chu	"Reactions of Lithium Dimethylcuprate with Diaryl Ketones"	Dr. H. O. House
Dr. M. Lusch	"Stereochemistry of Metal-Ammonia Reductions"	Dr. H. O. House
Dr. K. A. Snoble	"The Use of $\beta$ -Cyclopropyl $\alpha,\beta$ -Unsaturated Ketones"	Dr. H. O. House



IV. Research in Progress (Continued, Post-Doctoral Investigations)

<u>Name</u>	<u>Title of Research</u>	<u>Advisor</u>
Dr. M. Deutsch	"Photochemical Synthesis of Sesquiterpenes"	Dr. D. S. Caine
Dr. L. Gelbaum	"Natural Product Chemistry of Eupatorium Rugosom and S. Aureus"	Dr. L. Zalkow
Dr. M. Ghosal	"Natural Product Chemistry of Eupatorium Rugosom"	Dr. L. Zalkow
<u>Ph.D. Theses</u>		
Ms. Joyce Wilkins	"Reactions of Organocopper Compounds"	Dr. H. O. House
Mr. Edward Zaiko	"Synthesis of Gibberellins"	Dr. H. O. House
Mr. William Phillips	"Stereochemistry of Enolate Alkylation"	Dr. H. O. House
Mr. William McDaniel	"Synthesis of Gibberellins"	Dr. H. O. House
Mr. Nabih Ghali	"Synthesis of Polyphenyl Anthracenes"	Dr. H. O. House
Mr. Cheuk C. Yau	"A New Synthesis of Cyclic Ketones"	Dr. H. O. House
Ms. Gloria Mischuk	"Stereochemical Study of Grignard Reagents"	Dr. H. O. House
Mr. Ronald Sieloff	"Reactions of Organochromium Compounds"	Dr. H. O. House
Mr. J. J. Watkins	"Reactions of Organocuprates"	Dr. E. C. Ashby
Mr. S. A. Noding	"Reduction and Alkylation of Model Ketone Systems"	Dr. E. C. Ashby
Mr. K. D. Hodges	"Studies of Metal Complexes of Macrocyclic Tetramines"	Dr. E. K. Barefield
Mr. D. J. Sepelak	"Organometallic Chemistry of Carbon-Nitrogen Multiple Bonds"	Dr. E. K. Barefield
Mr. T. Maurer	"Synthesis of Thiazridines"	Dr. E. M. Burgess
Mr. M. S. Steltenkamp	"Studies of the Enzyme System P. Oleovorans"	Dr. S. W. May
Mr. P.-C. Lu	"Arrangements of $\omega$ -p-Biphenyl-alkyl Anions"	Dr. E. Grovenstein, Jr.

IV. Research in Progress (Continued, Ph.D. Theses)

<u>Name</u>	<u>Title of Research</u>	<u>Advisor</u>
Mr. J. H. C. Lee	"Applications of Partially Relaxed FT NMR to Chemical Analysis"	Dr. S. L. Gordon
Mr. S. A. Frobese	"Photochemical Synthesis of Sesquiterpenes"	Dr. D. S. Caine
Mr. Troy Smith	"Photochemical Synthesis of Sesquiterpenes"	Dr. D. S. Caine
Mr. W. Samuels	"Photochemical Synthesis of Sesquiterpenes"	Dr. D. S. Caine
Ms. M. Gordon	"Natural Product Chemistry of Daucus Carota"	Dr. L. Zalkow
Mr. J. Novak	"Natural Product Chemistry of Aplopappus Heterophyllis"	Dr. L. Zalkow
Mr. B. Ekpo	"Natural Product Chemistry of Aplopappus Heterophyllis"	Dr. L. Zalkow
Mr. R. Harris	"Natural Product Chemistry of Aplopappus Heterophyllis"	Dr. L. Zalkow
Mr. J. Baxter	"Natural Product Chemistry of Bothriochloa Interimidia"	Dr. L. Zalkow
Mr. T. Caruso	"Synthesis of Chiral Crowns"	Dr. C. L. Liotta
Mr. H. Colvin	"Synthesis of Sulfur Crowns"	Dr. C. L. Liotta
Mr. B. O'Brian	"Syntheses Involving New Oxidizing Reagents"	Dr. C. L. Liotta
Mr. A. Dabdoub	"Reactions of Naked Cyanide and Acetate"	Dr. C. L. Liotta
<u>M. S. Theses</u>		
Mr. E. J. Jeroski	"Arrangements of $\omega$ -Phenylalkyl Alkyl Anions"	Dr. E. Grovenstein, Jr.